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TEST PROGRAM OF GRANULAR MATERIALS FOR THE THIN-WALLED, PARTICULATE-PACKED CONTAINER

Report No 87-154-K
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ABSTRACT

Several immobilization container designs are being evaluated for disposal of used fuel bundles from the CANDU Nuclear Power Stations. Two of those designs have a thin titanium shell on the outside. The free space inside the containers will be filled with a granular material (particulate) which will provide the structural support for the thin external shell. The particulate must be compacted well to eliminate voids. It must have an adequate strength to support the postulated external pressure of 10 MPa, low dust content and low creep. An extensive test program was performed on several particulates to select the best available material. The program consisted of: pouring test and compaction test using sine-wave vibrations, uniaxial compression tests and creep tests. Dust content was observed during the tests. Most of the particulates performed well during all tests. They all have similar packing ability and strength. However, their dust content varies quite widely. Glass beads appear to be the most suitable particulate tested.

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EXECUTIVE SUMMARY

TEST PROGRAM OF GRANULAR MATERIALS FOR THE THIN-WALLED, PARTICULATE-PACKED CONTAINER

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The Thin-Walled, Particulate-Packed and Structurally Supported, Particulate-Packed containers were developed for immobilization and disposal of used fuel bundles from CANDU Nuclear Power Station in an up to 1000 m deep vault, in the Canadian shield. The containers will have a thin (4.76 to 6.35 mm thick) titanium shell on the outside. The shell will be supported by an internal matrix made of a granular material (particulate) which will fill all free space inside the container. The criteria used to select the particulate include: good pouring characteristics (to ensure that all voids are filled); adequate strength to support a postulated external pressure of 10 MPa; high modulus of elasticity to minimize plastic deformations in the titanium shell; low dust content to faciliate welding of the closure; low creep for the long term support; low cost and good availability. These properties were compared for nine particulates:

sand	glass beads	steel shot
bauxite	zircox	rutile-zircon-garnet mix
zircon	rutile	sintered bauxite

All particulates are readily available at an acceptably low cost. Except zircox 51702, they all have good packing properties, adequate stiffness and strength. Steel shot has the highest stiffness at 648 MPa. Stiffness of all other particulates ranged from 43 to 360 MPa. Dust content of the particulates ranged widely. Glass beads appear to be the most suitable particulate based on the test results.

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NOMENCLATURE

AECB	-	Atomic Energy Control Board
AECL	-	Atomic Energy of Canada Limited
Container	-	A receptacle for burying used nuclear waste
Disposal Facility	-	A facility used to emplace or discharge waste materials with no intention of retrieval (for example an underground disposal vault and its supporting facilities).
Disposal System	-	A disposal facility and the transportation facilities needed to bring the nuclear fuel wastes from the interim storage sites.
Ontario Hydro Container	-	Synonymous with Thin-Walled, Particulate-Packed Container
OHRD	-	Ontario Hydro Research Division
Particulate	-	Granular material such as glass beads or steel shots
TWPP Container	-	Thin-Walled, Particulate-Packed Container
Used fuel	-	Fuel extracted from a reactor at an end of its useful cycle.
WNRE	-	Whiteshell Nuclear Research Establishment

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To Mr. H. N. Isaac
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TEST PROGRAM OF GRANULAR MATERIALS FOR THE THIN-WALLED, PARTICULATE-PACKED CONTAINER

1.0 FUEL DISPOSAL CONCEPT

The Canadian used fuel disposal program management is provided by Atomic Energy of Canada Limited through their Whiteshell Nuclear Research Establishment Division (WNRE). Ontario Hydro Research Division (OHRD) provides assistance to WNRE in several areas of the program. The program is based on immobilization of unreprocessed fuel bundles in high integrity containers. The containers will be placed in an up to 1000 m deep repository located in the plutonic rock of the Canadian shield. A schematic of the repository is shown in Figure 1/1/.

The containers are designed to survive at least 500 years without leaking, in the adverse conditions of the repository. Several alternative container designs are being investigated. Two of those designs rely on a granular material (particulate) for their internal support:

- The Thin-Walled, Particulate-Packed (TWPP) container shown in Figure 2.
- The structurally-supported particulate-packed container shown in Figure 3.

WNRE is placing more emphasis on the first design, presently. The specific requirements of this design are considered here in detail.

2.0 CONTAINER DESCRIPTION

The container is designed to operate under the following conditions:

- A minimum external hydrostatic pressure of 10 MPa which could exist in a flooded, 1000-m deep disposal vault.
- A maximum container skin temperature of 150°C.

A thin-walled container shell cannot support the postulated high external pressure. The titanium shell is designed to be supported by a carefully packed particulate. Stiffness of a particulate in the free state is much lower than the modulus of elasticity of titanium, but it increases rapidly when external pressure is applied. Small compressive, plastic deformation of the titanium shell is anticipated. Such deformation should not jeopardize the integrity of the container, because of the good plastic properties of titanium/2,3/. However, poorly packed particulate or excessive gaps, such as between the top surface of the particulate and the top lid could result in large deformations of the shell and in failure of the container's integrity.

The TWPP container consists of a carbon-steel basket that rests inside a cylindrical titanium shell. The basket retains 72 used-fuel bundles while allowing for the free flow of particulate during packing and compaction. The basket consists of nineteen, 4-in Sch. 10 tubes concentrically arranged as illustrated in Figure 4. A spoked wheel arrangement (see Figure 4) at the bottom of the basket supports the bundles inside the pipes, while allowing for a free flow of particulate.

The outer shell is made from 4.76 mm thick, rolled ASTM B 265 grade 2 titanium plate. It is about 2246-mm high and has a diameter of about 630 mm, see Figure 2. A 6.36-mm thick bottom head is welded to the shell. The top lid is 4.76 mm thick and will be diffusion bonded to the shell after assembly. The larger thickness of the bottom plate is required to support the weight of the particulate and filled basket during lifting. Empty space inside the container is filled with a granular material compacted by vibration to form the structural matrix.

3.0 PARTICULATE REQUIREMENTS

The particulate must satisfy the following conditions for the mechanical stability of the container:

- fill all voids without clogging;
- be small enough to flow between the fuel bundle elements (less than 1.2 mm diameter) but the grains should be heavy enough to avoid becoming airborne during vibratory compaction.
- have sufficient strength to withstand a pressure of 20 MPa (including a safety factor of 2)
- have adequate stiffness to prevent large plastic deformations of the titanium shell
- have low dust content to minimize airborne particles
- should not adhere to the container wall, to simplify welding of top lid
- have small creep deformations over the 500-year container life

- have sufficiently high bulk modulus under external pressure.

4.0 TESTED PARTICULATES

Eleven particulates were selected for the test program which represent the wide range of materials considered/4/. They include:

- Wedron sand, type 4098
- fine glass beads size 0.002 mm to 0.3 mm
- glass beads size 0.8 mm to 1.2 mm
- steel shot
- aluminium oxide powder
- crashed bauxite grains
- sintered bauxite
- Interprop
- Zircox 51702
- mixture of 38% rutile, 38% zircon and 24% garnet (R-Z-G)
- zircon
- rutile.

A typical chemical composition of the tested particulates is shown in Table I. Tests of the first four particulates were described in the Atomic Energy of Canada Limited Technical Record TR-131/5/. For completeness, all relevant information from that report is also presented here.

5.0 TEST PROGRAM

The particulates underwent a two stage test program. In the first test, a particulate was poured into a plexiglass tube containing two simulated Bruce bundles. During pouring, dust formation was observed. After examination for voids, sine-wave vibration was used to compact the particulate further.

The compacted particulate was then subjected to uniaxial compression. From this test, the mechanical properties of the particulate were estimated. These included: Young's modulus of elasticity, bulk modulus and Poisson's ratio. Compression was continued until the grains of particulate started to fracture. This point was designated the particulate strength level.

Uniaxial creep tests were conducted for up to 42 days to determine the stability of particulate under external pressure.

6.0 VIBRATORY COMPACTION TESTS

6.1 Test Equipment

The first six particulates listed in section 4.0 were tested in 1980/1/, the rest in 1986. Although the shaker table arrangement was different in the two sets of tests, the particulate compaction equipment was similar. The test equipment used in the original and subsequent particulate compaction tests is shown in Figure 5. The pouring and vibratory compaction tests took place in a 106 mm diameter plexiglass tube 1092 mm long. The tube contained two simulated Bruce bundles. The centre pencil of each bundle was removed and, in some of the original tests, replaced with a 1143 mm long, 16 mm diameter plexiglass tube (see Figure 5). This centre tube was used with fibreoptics equipment to observe if voids formed near the centre.

6.2 Objectives

The vibratory compaction tests were performed to assess the packing properties of various particulates. Simulated fuel bundles were assumed to resist the flow of particulate. To evaluate this resistance, three different diameter plexiglass tubes were prepared having different inside diameter (different gap size between the bundles and tube wall).

Different accelerations, frequencies and amplitudes of displacement were used to determine the optimal packing conditions.

The formation of dust was observed during pouring of particulate.

6.3 Procedure

A plexiglass tube with 2 simulated bundles and the plexiglass centre tube (used in the first few tests only) was placed inside the channel frame and mounted with 4 brackets. The frame was connected to an actuator system. Equipment prepared for testing is shown in Figure 6a. As particulate is poured in, it settles to the bottom of the tube, in most cases, as shown in Figure 6b. While the system remained stationary, particulate was poured into the tube until it covered the bundles (see Figure 6c). Sine-wave vibrations were then introduced at a selected acceleration and frequency for 5 minutes. In most cases, the 5 minutes vibratory compaction was repeated at the same or different accelerations. The time limit of 5 minutes was arbitrarily selected. The second 5 minutes compaction usually contributed less than 10% of the total reduction in volume due to compaction. The level of particulate after compaction is shown in Figure 6d.

The weight and volume of particulate were recorded before and after each period of vibrations. The derived densities of particulate were compared with the "reference density" (see next section).

The effect of two different orientations of the two bundles in the tube were tested. Figure 7 shows pencils of the bundles in an out-of-line position. An in-line arrangement produced identical results.

Only the first test was made at 2 g peak-to-peak acceleration. All subsequent tests were conducted at 4 g, since higher accelerations improve packing characteristics. All particulates were moisture free prior to the tests.

6.4 Reference Density

The reference density is defined as the theoretical largest density to which spherical beads of equal size can be packed. The largest density of packing is obtained when face-centred-cubic orientation of beads is achieved. This orientation is shown in Figure 8. The efficiency of packing is equal to the ratio of the volume occupied be the spheres to the volume of the tube, or:

$$e = Vb/Vt = 5.333\pi\ (r/a)^3$$

where

Vb - volume of spheres inside the cube in Figure 8
Vt - volume of the cube
$a = 2\sqrt{2}\ r$ (see Figure 8)

$$e = 0.74048$$

Density of a particulate is initially calculated by submerging a known weight of particulate in water and monitoring the increase in volume of water. The calculated density is multiplied by e to obtain the reference density.

Reference density = 0.74 x solid block of particulate material

The compaction ratio is a ratio of the largest density obtained in a set of tests over the reference density for the given particulate.

6.5 Results

The results of the vibratory compaction tests are given in Table II.

In most cases, the bundles formed no significant resistance to the flow of particulate. The relative orientation of the two bundles (see Figure 7) did not have any noticeable effect either. All particulates settled to the bottom densely during pouring (except zircox 51702). If we use the ratio of achieved density to the reference density as a measure of particulate performance, rutile, interprop, sintered bauxite rutile-zircon-garnet mixture and glass beads performed well. During the glass bead and sintered bauxite tests, the as-poured density has not increased much in the subsequent vibrations. This might indicate that these two particulate can settle faster with less shaking.

Following pouring of particulate a visual inspection was made for localized voids. Voids were not observed at the outside tube or at the centre tube during all tests, except for the Zircox 51702 test. During Zircox tests several large voids formed (see Figure 9). A fibreoptics device was used to

see if voids form in the centre of the tube. Voids were not seen in the first few tests. The centre tube was abandoned in subsequent tests.

Following vibratory compaction, the top surface of particulate was observed to be inclined at an angle of up to 30°C. This occurred in many tests but no effort was made to explain it. The largest slope appeared to form for particulate having rough surfaces, such as sand and Zircox. In smooth particulates such as glass beads and sintered bauxide, the angle was small. The slope in the sand is illustrated in Figure 10.

From test 3 (in Table II), it can be seen that the density obtained during the initial step of vibratory compaction at 150 Hz did not increase in the following step at lower frequency. However significant improvement was observed in test 4 after frequency was increased from 25 to 90 Hz. Further increase to 180 Hz did not improve the density. These tests indicate that a frequency of 50 to 90 Hz and a highest possible acceleration are the most beneficial. Vibration should continue for a long time as we learned during the prototype test program, discussed in Reference 2.

A prototype of the TWPP container was built and assembled at the Ontario Hydro Research Division facilities in Toronto. Glass beads were used as particulate. 700 kg of particulate were used to fill the void space of about 420 liters inside the container. The particulate was compacted using sine-wave vibrations with accelerations ranging from 1 to 4 g peak-to-peak for the total of 15 minutes. The obtained density of glass was only 1.67 kg/liter (reference density is 2.33 kg/l). After full assembly, the container was shipped by truck to the Whiteshell Research laboratories in Whiteshell, Manitoba. During the 30-hour travel, we estimate that the particulate settled further by about 4.4 liters to the density of 1.68 kg/l. This settlement resulted in an up to 14.5-mm high void at the top of the container. A 1.05% reduction in the volume of particulate resulted in a large void. Settlement of particulate of 0.33% should be considered significant. A detailed discussion of the 14.5 mm void and of the required packing quality can be found in reference/7/.

The formation of dust was observed during pouring of particulate into the tube. The volume of dust formed during pouring of aluminium oxide powder was excessive and it inhibited the tests. The least dust was observed during pouring of steel shot and glass beads. Dust content of all particulates is given ranking from 0 (no dust) to 5 (dust inhibits testing) and is shown in Table II.

Of all tested particulates, steel shot, glass beads, interprop and sintered bauxide are the easiest particulates to pack. We conclude that Zircox 51702 should not be used because of its tendency to form voids. Sand, zircon, rutile, zircox and rutile-zircon-garnet combination should not be used because of their high dust content.

7.0 COMPRESSION TEST

7.1 Test Equipment

A cylinder 171.5 mm (6.75 inches) long is used for the tests (see Figure 11). The cylinder is made of a standard 4 inch, schedule 80 pipe. The pipe rested on a rubber pad and was closed with two pistons, one at each end to ensure that the external pressure was applied from both top and bottom of the tube and to minimize the pressure variation in particulate (see Figure 13).

The effect of a bundle on the properties of particulate was evaluated using a 65 cm high cylinder made of 5 inch schedule 160 pipe. A one-inch plate was welded at the bottom of the pipe. The top of the pipe was closed with a large piston. This equipment is shown in Figure 12.

7.2 Objectives

The tests were conducted to determine the structural properties of particulate including strength of beads to breakdown (elastic limit), moduli of elasticity and Poisson's ratio. Those properties of particulate are required to determine their ability to support the titanium shell. The effect of fuel bundles on the modulus of elasticity and Poisson's ratio were also estimated.

7.3 Procedure

A particulate was poured into the cylinder and placed under an universal compression machine. The force was applied through the top piston. Strain-gauges mounted on the side of the cylindrical shell were monitored to determine the pressure distribution in the cylinder. From the applied pressure, stress distribution in the cylinder and distance by which the piston was pressed in, the following properties of particulate were calculated:

- Young's modulus of elasticity
- Shear modulus of elasticity
- Poisson's ratio
- Bulk modulus of elasticity.

Strength of particulate was determined by two techniques:

- stress at which beads start to fail in large quantities, seen in glass beads (see Figure 14)

- stress at which Young's modulus becomes near zero.

Glass beads and sand were tested at elevated temperatures of 150°C to 200°C. The effect of elevated temperature on other particulates was evaluated in the creep test program.

7.4 Results

Following each test, the plugs were removed from the cylinder by hand without difficulty. The plugs were covered by a thin layer of dust following each test, due to particle crushing. A thin layer of crushed particulate formed crust at the top and bottom of the container, at pressures above 100 MPa, where such pressure was achieved.

The elastic limit of a particulate was defined as the point where a large volume of particle failures begin to occur. In most particulates tested such point could not be identified because the rate of particle failure was uniform throughout the test. However, if this uniform rate was high and, as pressure increased, Young's modulus dropped to near zero, the corresponding pressure is reported as the elastic limit.

Calculation of the four parameters was based on the stresses in the cylindrical shell measured by strain-gauges shown in Figure 11. The method of calculation is discussed in Appendix A. Stresses in the cylindrical shell are not uniform indicating large pressure variation with height in a particulate.

Relationship between the applied force and the piston deformation was plotted. All particulates show some breaking and settling of particles, starting at a very low pressure, as shown in Figure 14, by the difference between applying and releasing of the load. The results from the test are summarized in Table III.

For a safety factor of two, the strength must not be less than 20 MPa, or twice the repository pressure. Use of crushed bauxite, rutile-zircon-garnet combination and zircox is not recommended because their strength is too low. Zircon and rutile are not recommended because their moduli of elasticity are too low, at below 250 MPa.

Bundles increase the stiffness of the particulate more than three times (see Table III). This effect has only a secondary importance because the basket tubes provide more stiffness than we expect from the particulate-bundle combination, as shown in the stress analysis of the container/2/.

The results of the high temperature tests showed a slight drop in the strength of most particulates. In glass beads, strength increased at elevated temperature. This increase is attributed to the reduced brittleness in particulate and fewer particle breaks. The breakdown point in glass beads becomes less prominent at higher temperature, see Figure 15.

The results presented above are only approximate. Further study may be required to confirm the properties listed in Table III. The test on glass beads, used to pack the prototype, was conducted twice. The obtained moduli of elasticity ranged from 337 MPa (obtained in the first test) to 360 MPa

obtained in the second test. The results obtained during testing of zircox 51702 were even more disturbing because Poisson ratio of over 0.5 was determined in the first test. Such a Poisson's ratio is technically impossible. Results from only the second test were used. For reference, the results from both tests are shown in Table IV.

8.0 CREEP TEST

The container must survive 500 years without penetrations. The selected particulate must be adequately stable to provide the required support. Creep tests were conducted to determine if the particulates would deform under long-term pressure of 10 MPa. Two particulates were tested simultaneously.

8.1 Test Equipment

Two 27-inch-high cylinders were mounted in a frame shown in Figure 16. The cylinders were made of standard 3 inch schedule 160 pipes closed at the bottom with a plate welded in place. A hydraulic system was used to apply a constant pressure of 10 MPa. A pneumatic pump generated the pressure. The cylinders had heaters strapped to them and were insulated, as shown in Figure 17. One tube had strain-gauges (see Figure 17). Before actual, high temperature tests started, the axial stress distribution in the particulate was determined.

8.2 Objectives

Creep behaviour of various properties was evaluated in an up to 1000 hour test. Creep equations were derived and used to extrapolate the available data and to predict the total 500 year creep deformation.

8.3 Background Information

Ideally, creep in solids is divided into three stages. The primary or logarithmic creep diminishes with time. The creep rate is quite fast at the beginning and slows down until it stops for all practical applications. Before the creep rate reaches zero, a second stage may initiate where constant creep rate dominates. In tension, this stage ends when the material weakens and rate of creep increases. The third stage of creep is characterized by continuously increasing creep rate until failure. The idealized creep diagram is shown in Figure 18.

Creep rate is commonly described by an equation:

$$\dot{\varepsilon} = A\, S^n\, t^m\, \varepsilon^{rT}$$

where

- A - equation constant
- S - stress level
- t - time
- T - temperature
- n - creep stress exponent
- m - creep time exponent $m \geq -1$
- r - creep temperature exponent $r \leq 0$

Value of m determines the stage of creep:

in the primary creep $-1 \leq m < 0$
in the logarithmic creep $m = -1$,
in the steady state creep $m = 0$
in tertiary creep $m > 0$

Strain in the first stage of creep is frequently given by a logarithmic line in form

$$\varepsilon = A + B \ln(t)$$

The second stage is described by a linear equation:

$$\varepsilon = A + B\, t$$

In both equations, A is the initial (plastic) deformation and B indicates the rate of settlement. The rate of settlement is larger for larger B.

The above consideration of the idealized creep curve is valid only for solid materials under tensile loading. The particulate is always in compression. Although all three stages are possible in a particulate in the initially, deformation can only continue until crust forms or internal free space is very small (very large forces are required to induce any additional deformation). At this stage, further deformations are not possible. However, this deformation could be high with the final volume equal to the ratio of the density of particulate over the density of solid block of material from which particulate was made.

8.4 Creep Test Procedure

The particulate was poured into a cylinder within 30 mm from the top. The particulate was compacted by impacting the cylinder with a rubber mallet for about 5 minutes. The final level of particulate was about 50 mm from top. The hydraulic system was connected but not pressurized. The heaters were turned on. The temperature of particulate was maintained at 150°C during the tests. Eighteen hours after the heaters were turned on, the pressure was applied. The tests continued for 15 to 42 days. Travel of the piston as a function of time was plotted (see Figure 19).

Several additional tests were made on glass beads, considered as the reference particulate at this stage. These tests included:

- multiple pressure fluctuation from 0 to 10 MPa at constant temperature,
- multiple temperature fluctuations at constant pressure,
- an evaluation of the effect of distilled, di-ionized water on creep in glass beads,

- an effect of Standard Canadian Shield Saline Solution on the performance of glass beads.

The last two tests were conducted at two temperatures of 22°C and 75°C each.

8.5 Results

Most of the particulates performed well during the tests. In the evaluation of the results, the assumption was made that the creep will be in its first stage. The experimental results were then compared with a logarithmic curve to confirm this assumption.

Figure 19 shows results for five particulates which confirm the above assumption well. They include sand, zircon, R-Z-G combination, rutile and Zircox 51702. In glass beads, Interprop and sintered bauxite the early settlement was slower than a logarithmic curve would indicate (see Figure 20). Logarithmic relationship is used because it approximates well the deformations in the later parts of tests.

The results of tests on creep in steel shot showed that the strain varied nearly linearly with time indicating secondary (or even tertiary) creep. Creep in steel can occur at above 100°C and near yield stress/8/. Stresses in steel shots are high due to the contact (Hertz) effects. Recent creep tests further confirmed the results from reference/8/ in reference/7/. Steel shot should not be used as packing material.

Coefficients of the creep equations have been evaluated for all tested particulates and are listed in Table IV. The creep equations were used to estimate the 500-year creep strain which would occur if the trend established in the tests continued for 500 years. The obtained rate of deformation in Zircox 51702 is high. This particulate should not be used. In several cases, the experimental results are above the logarithmic curve, in the middle of a test. This indicates that the long term logarithmic estimate of strain may be conservative. This includes such particulates as glass beads and interprop, shown in Figure 20.

Large settlement was observed during initial hours of most tests. This is attributed to inadequate packing of particulate. Better compaction should be obtained in the disposal program. In Table V, the strains measured during the first 100 hours is separated from the estimated 500-year creep to distinguish between the initial and long term settlements.

Glass Beads are considered the "reference particulate". Their test program was extended to determine their performance in the presence of two liquids: distilled, de-ionized water and Standard Canadian Shield Saline Solution (SCSSS). SCSSS composition is given in Table VII. Results of the test is shown in Figure 20. The test was conducted at 22°C, initially. After 1000 hours, the test was stopped. The beads in the SCSSS were examined and found undamaged. The particulate was then heated to 75°C and the tests resumed.

The results of the dry glass bead test are included in Figure 21 for comparison. Performance of glass beads in both solutions is similar. The settlememt obtained in liquids was less than during the dry bead test. The obtained results could be caused by different quality of compaction before the test was started or density of particle improved during pouring of liquids.

The results of test program are expected to have limited accuracy. Creep tests are generally known to have large scatter of results. The results described above should be accepted as indications of particulate behaviour rather than accurate measurements of true property. We have not determined the nature of the creep deformation although several mechanisms have been postulated:

- Creep is due to continuous rearranging of particles which reduces their total volume

- A few highly stressed particles crack periodically to facilitate rearranging of other particles

- The material from which the particle is made creeps.

The first two sources of creep are assumed to be dominant in all tested particulates except steel shot. Strain-gauges were used before tests started to determine the axial stress variation in the particulate. Local hoop stress in the cylindrical shell was assumed to indicate the stress level in particulate at that elevation. The variation in hoop stress is large as shown in Figure 22. We assume that after a 1000-hour test this variation would be reduced. We were not able to confirm this hypothesis because the gauges were damaged at the end of the test.

Figure 23 shows the results of pressure and temperature fluctuations on the settlement of glass beads. The first two pressure (or temperature) cycles caused larger settlement than the 500 year steady state settlement discussed above. The settlement caused by cycling pressure is not due to creep in the particulate material but due to rearrangement of beads.

9.0 SUMMARY

An extensive test program has been performed on nine different particulate materials to determine if they can provide an adequate structural support for the Particulate-Packed containers. Three of those particulates are considered adequate. We feel that further testing would be highly beneficial as it could provide more reliable results for the three particulates and to allow us to set preferences.

9.1 Comparison of Results

Based on the completed tests, only three particulates are considered viable: glass beads, interprop and sintered bauxite. All other particulates were disqualified during the compression test, except steel shot. Zircox 51702 was considered unsuitable in all tests. Compaction tests showed an excessive dust content in rutile-zircon-garnet combination, sand, zircon, zircox and rutile. Steel shot were disqualified because of their low-temperature creep.

Glass beads appear to have the lowest dust content, the best packing performance and the highest modulus of elasticity of the three recommended particulates. However, they have a clear breakdown point which limits their strength to 27 MPa. Their breakdown point and modulus of elasticity could be improved further by proper selection of their composition. Interprop and sintered bauxite have much higher strength then glass beads, of above 50 MPa. Sintered bauxite has the lowest modulus of elasticity at 285 MPa.

9.2 Further Tests

Most of the tested particulates performed well during all tests. Preferences of one particulate over another was frequently made based on subjective judgement (dust content observation) or on differences between results which could be within the accuracy of a test (moduli of elasticity of the three preferred particulates).

The author recommends that further tests could provide a better distinction between the three particulates.

Other tests could be added to further distinguish between the prospective particulates, such as: heat transfer properties, stability during welding and in high radiation field, chemical stability and so on. Dust content could be quantified. Performance of particulate at elevated temperature should be evaluated more thoroughly.

The particulate test program is still far from completion, although some preferences are justified at this stage.

10.0 CONCLUSIONS

In the limited scope of tests conducted to date, glass beads should be considered to be the most suitable particulate tested. Two other particulates have been shown viable: interprop and sintered bauxite. However, many test results are considered inconclusive due to their limited accuracy. We recommend that the program should be continued.

11.0 ACKNOWLEDGEMENTS

Tests were conducted by Messers J. Russell, A. Fallis and R. Zane. WNRE personnel conducted a critical review of the manuscript. Mr. L. Hosaluk of WNRE contributed to Appendix A. The author acknowledges and thanks them for their input.

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REFERENCES

1. "A Disposal Centre for Irradiate Nuclear Fuel: Conceptual Design Study", AECL Technical report AECL-6415, prepared by Acres Consulting Engineers Limited and Dilworth, Secord, Meagher and Associates, Dated September, 1980.

2. B. Teper, "Preliminary Evaluation of Stress-Strain Distribution in Particulate-Packed Container", Ontario Hydro Research Division Report 825.94, issued October 29, 1981.

3. B. Teper, "Test Program of the Prototype of the TWPP container, Part 1 - Hydrostatic Test", Ontario Hydro Research Division Report No. 84-293-K, Issued December 21, 1984.

4. K.N. Shelson, "Particulate Packings for Irradiated Fuel Disposal Containers", Ontario Hydro Design and Development Division Report 83085, dated January 1983.

5. B. Teper, "Particulate Compaction Tests for a Particulate-Packed, Thin-Wall Container for Irradiate Fuel Disposal", AECL technical Record TR-131, dated December, 1980.

6. "Soil Mechanics", I.W. Lambe and R.V. Whitman, John Wiley and Sons Inc, 1969 (chapter 12).

7. B. Teper, "Test Program of the Prototype of the TWPP Container, Part 3 - Detailed Stress Analysis", to be issued as a Ontario Hydro Research Division Report soon.

8. B. Teper, "Evaluation of Iron-Based Stressed-Shell Container Concept", Ontario Hydro Research Division Report 86-98-K, dated August 5, 1986.

9. B. Teper, S. Reid, "Feasibility Assessment of the Iron - Based, Stressed-Shell Container", to be issued as a Ontario Hydro Research Division Report soon.

TABLE I

TYPICAL COMPOSITION OF PARTICULATE

PARTICULATE TYPE	% WEIGHT									GRAIN SIZE
	SiO_2	AL_2O_3	TiO_2	Fe_2O_3	MgO	NaO	CaO	ZrO_2	HfO_2	mm
WEDRON SAND	99.9	0.1								0.5 - 1.2
GLASS	37.8					0.9	14.1			0.5 - 1.0
BAUXITE GRAINS	2.7	87.0	4.3	5.6	0.2		0.2			
SINTERED BAUXITE	2.7	87.0	4.3	5.6	0.2		0.2			0.1 - 0.4
INTERPROP	15.5	74.0	3.7	5.5	0.3			0.5		0.3 - 0.5
ZIRCOX	1.8	0.2	1.2	0.1			2.1	53.8	40.7	0.2 - 2.0
GARNET	38.0	26.0	1.0	30.0	2.0		1.0	1.0		
RUTILE	2.5	0.75	93.0	1.5				2.0		0.7 - 0.25
ZIRCON	15.7	0.6	1.3	0.3			1.5	50.2	30.3	0.06 - 0.15
MIXTURE	16.0	6.7	36.1	7.9			0.8	20.1	11.7	0.6 - 1.0

TABLE II
VIBRATORY COMPACTION TEST RESULTS

TEST NO.	TYPE OF PARTICULATE	AMOUNT POURED (kg)	VOLUME POURED (L)	REFERENCE DENSITY ρr(kg/L)	$(\rho/\rho r)$ MAX (%)	DENSITY OF POURING (kg/L)	FIRST FREQ (Hz)	ACCEL (g)	VOLUME (L)	DENSITY ρ(kg/L)	SECOND FREQ (Hz)	ACCEL (g)	VOLUME (L)	DENSITY ρ(kg/L)	THIRD FREQ (Hz)	DENSITY ρ(kg/L)	SEPARATION OF SIZES	DUST
1	SAND-BUNDLES NOT INCLUDED	6.65	4.30	1.932	87.0	1.55	60	4	3.95	1.680							-	
2	SAND	7.52	4.83	1.932	89.1	1.56	30	4	4.40	1.710	60	4	4.30	1.715	120	1.72	-	3
3	SAND	6.76	4.36	1.932	89.0	1.55	150	4	3.92	1.72	70	4	3.92	1.72	25	1.72	-	
4	FINE GLASS (0.002 0.3mm dia)	6.60	4.12	1.780	97.8	1.60	25	4	3.89	1.70	90	4	3.80	1.73	180	1.74	LAYERS	3
5	FINE GLASS (0.002 0.3mm dia)	6.62	4.11	1.780	98.9	1.61	100	4	3.79	1.75	45	4	3.77	1.76			LAYERS	
6	GLASS BEADS (0.8 1.2mm dia)	8.17	4.20	2.221	91.4	1.94	40	4	4.04	2.02	110	4	4.02	2.03			-	1
7	A_2O_3 POWDER	TEST ABANDONED DUE TO EXCESSIVE DUST FORMATION																5
8	SINTERED BAUXITE	11.63	5.22	2.90	82.1	2.23	50	2	4.97	2.34	50	4	4.88	2.38				2
9	INTERPROP	8.69	4.75	2.38	83.6	1.83	50	2	4.39	1.98	50	4	4.36	1.99				2
10	ZIRCON	13.08	4.75	4.04	77.2	2.75	50	2	4.42	2.96	50	4	4.19	3.12				3
11	RUTILE	11.76	4.79	2.85	95.4	2.46	50	2	4.70	2.69	50	4	4.64	2.73				3
12	R-Z-G[2]	8.98	5.65	2.35	80.4	1.59	50	2	4.86	1.85	50	4	4.75	1.89				4
13	ZIRCOX S1702	12.01	6.37	4.00	65.8	1.89	50	2	5.96	2.02	50	4	4.57	2.63			VOIDS	3
14	GLASS BEADS	8.53	4.79	2.33	81.8	1.78	50	2	4.57	1.86	50	4	4.51	1.89				1
15	STEEL SHOT 0.8 - 1.2 mm	22.5	5.15	7.79	60.0	4.46	50	2	4.90	4.68	5	4	4.88	4.70				0

1. DUST IS RATED ON A SCALE FROM 0 (NO DUST) TO 5 (DUST CLOUD)
2. RUTILE - ZIRCON - GARNET COMBINATION
3. ACCELERATION IS GIVEN AS A PEAK-TO-PEAK VALUE

TABLE III

PROPERTIES OF PARTICULATES

NO.	PARTICULATE OR SOLID MATERIAL	TEST PRESSURE MPa	ν	E_p GPa	β GPa	E_p/E_t (1)	STRENGTH (2) δu (MPa)
	PARTICULATES						
1	WEDRON SAND	12.0	0.32	457	423	0.0042	*
2	GLASS BEADS 0.6-0.9	12.0	0.40	283	472	0.0026	32
3	GLASS BEADS 1.0-1.2	12.0	0.35	297	330	0.0027	27
4	GLASS BEADS 1.4-1.68	12.0	0.40	303	505	0.0028	27
5	PROTOTYPE GLASS BEADS	12.0	0.41	337	624	0.0031	27
6	STEEL SHOTS	12.0	0.29	5,615	4,455	0.0065	*
7	CRASHED BAUXITE	12.0	0.33	83	83	0.0008	14
8	INTERPROP	10.0	0.30	336	281	0.0031	*
9	SINTERED BAUXITE	10.0	0.34	285	297	0.0026	*
10	RUTILE	10.0	0.33	238	233	0.0022	*
11	ZIRCON	10.0	0.41	152	282	0.0014	*
12	ZIRCOX 51702	10.0	0.30	36	30	0.0003	12
13	COMBINATION R-Z-G	10.0	0.31	46	40	0.0004	14
	PARTICULATE WITH BUNDLE						
8	WEDRON SAND	9.7	0.26	1,662	1,151	0.0166	
9	GLASS BEADS 0.6-0.9	9.7	0.18	2,337	1,220	0.0234	
10	GLASS BEADS 1.0-1.2	9.7	0.34	1,041	1,055	0.0104	
	SOLID MATERIALS						
11	STEEL	--	0.29	200,000	160,000	2.00	
12	TITANIUM	--	0.34	100,000	110,000	1.00	
13	GLASS(3)	--	0.20	69,000	38,000	0.69	
14	QUARTZITE	--	0.14	82,700	38,300	0.83	
	PARTICULATE - LAMBE AND WHITMAN DATA (4)						
15	SAND	--	0.2-0.5	103	--	0.001	

(1) E_t = 100,000 MPa - Young's modulus of titanium

(2) Asterisk designates strength of above 50 MPa or clear breakdown point was not observed.

(3) The shown values are for illustration only as they vary widely with composition and treatment.

(4) Values quated in Reference 4 for pressure of up to 0.2 MPa.

TABLE IV

COMPRESSION TEST OF ZIRCOX

TEST 2

LOAD (LBS)	ν	E_p (MPa)	β (MPa)	δ (MPa)	TH AUG (MPa)	TA AUG (MPa)
4,200	.08	170	68	78	4.38	9.19
8,500	.18	85	44	36	9.2	8.24
11,300	.22	61	36	25	11.5	7.83
16,700	.30	36	30	14	15.5	6.98
24,000	.47	5	27	2	23.9	5.15
113,400	.31	95	81	36	96	41.9

TEST 1

LOAD (LBS)	ν	E_p (MPa)	β (MPa)	δ (MPa)	TH AUG (MPa)	TA AUG (MPa)
4,200	.19	130	69	55	9.34	7.8
8,500	.81	-55	29	-15	-19.5	.88
11,300	.44	14	43	5	23.9	5.73
16,700	.60	-21	35	-6	30.5	3.93
17,000	.60	-22	35	-7	33	4.15
24,900	.83	-67	34	-18	46.1	1.87

TABLE V

CREEP TEST RESULTS

PARTICULATE	STRAIN ($X10^6$)			CREEP EQUATION COEFFICIENTS (1)	
	FIRST 100 hr	100 hr 500 YEARS	0 TO 500 YEARS	A $X10^6$	B $X10^6$
GLASS BEADS	120	127	247	76.1	11.1
INTERPROP	50	136	186	22.9	10.7
SAND	167	54	221	144.3	5.0
ZIRCON	121	105	226	75.9	9.8
R-Z-G	69	96	165	33.6	8.6
RUTILE	271	91	362	237.3	8.2
SINTERED BAUXITE	33	144	177	5.0	11.3
ZIRCOX 51702	5,600	15,369	20,969	-1,150.3	1,471.3
STEEL SHOTS (2)	27			0.0	0.16
GLASS BEADS IN DDW(3)	74	69	143	10.55	1.65
GLASS BEADS IN SCSSS(4)	66	86	152	6.51	2.07

Notes:

1. Logarithmic distribution is assumed for all particulates except steel shots: Strain = A + B in (time)
2. Test indicates possibility of secondary creep in steel shots at 150°C. The equation used is:

   Strain = A + B x (time)

   Long term estimate is not realistic
3. Distilled de-ionized water
4. Standard Canadian Shield Saline Solution.

TABLE VI

COMPOSITION OF STANDARD CANADIAN SHIELD SALINE SOLUTION (SCSSS)

Ion	SCSSS
Na	5,050
K	50
Mg	200
Ca	15,000
Sr	20
Fe	-
Si	15
HCO_3	10
Cl	34,260
SO_4	790
NO_3	50
F	-
pH	7.0 ± 0.5

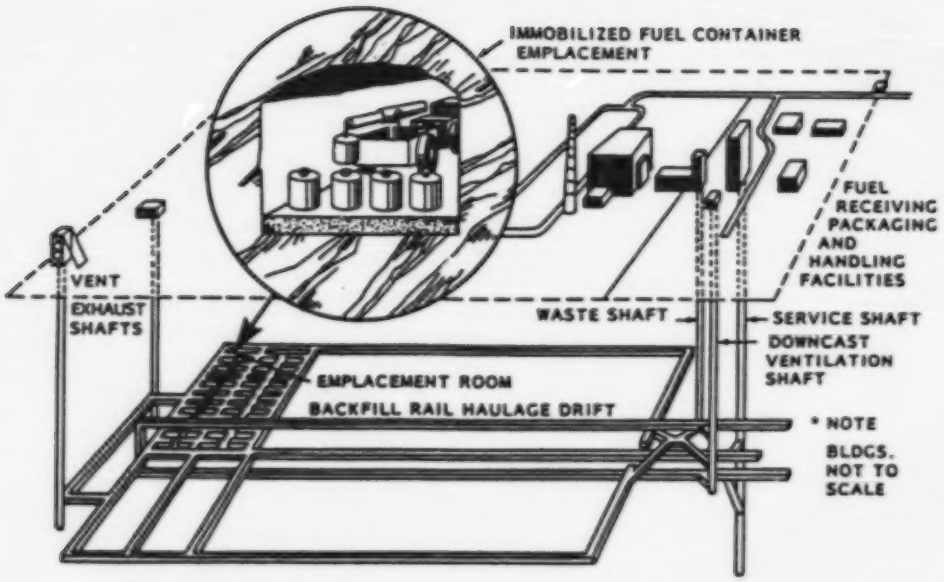


FIGURE 1
DISPOSAL FACILITY

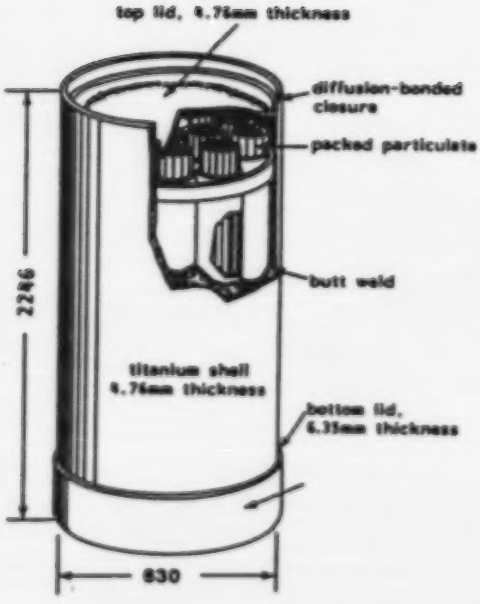


FIGURE 2
THIN-WALLED PARTICULATE-PACKED CONTAINER

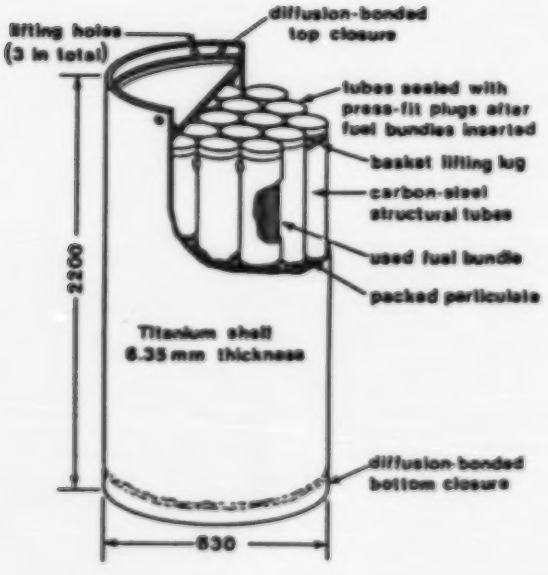


FIGURE 3
STRUCTURALLY SUPPORTED CONTAINER

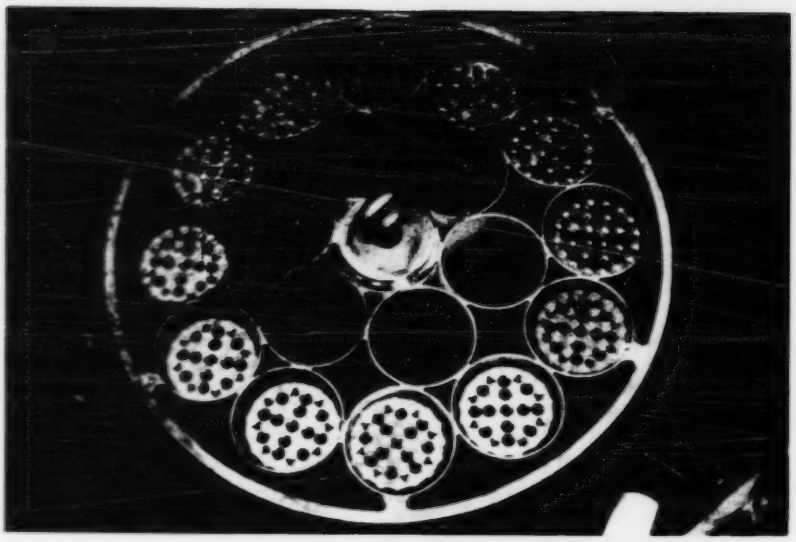
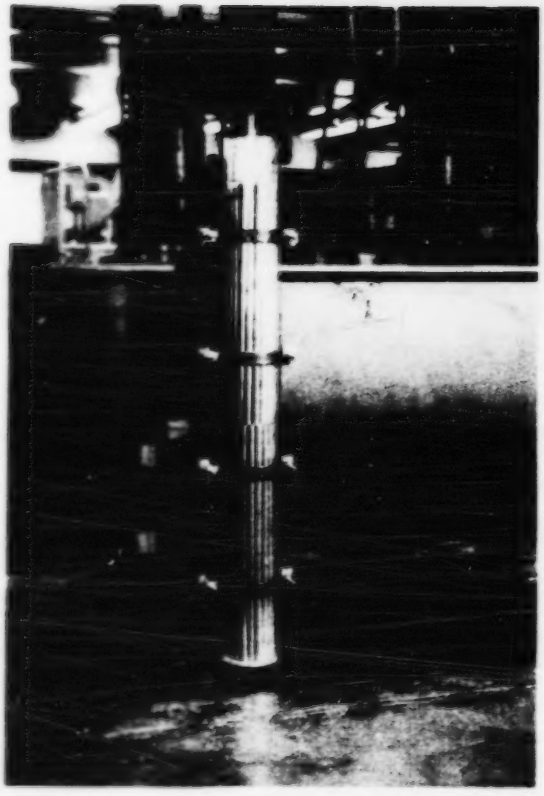
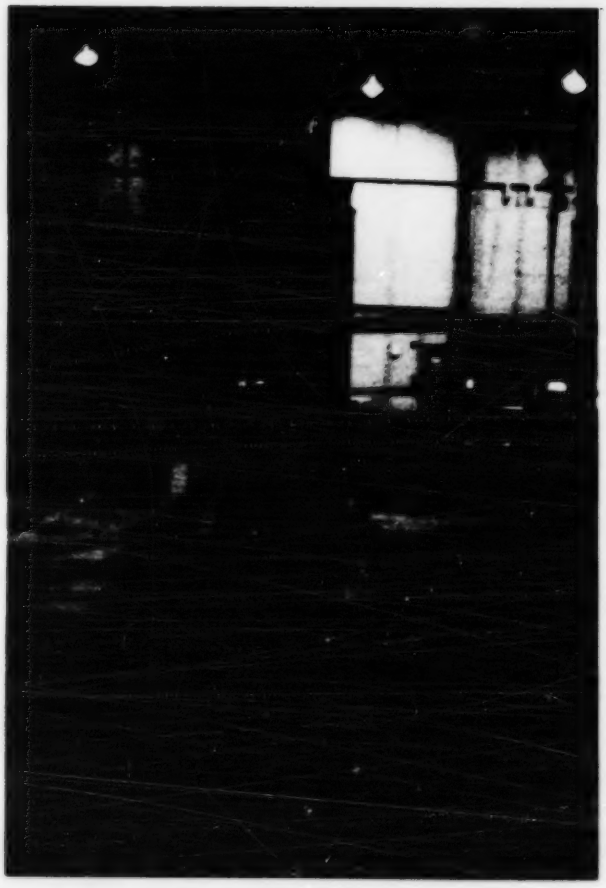


FIGURE 4
BASKET ARRANGEMENT

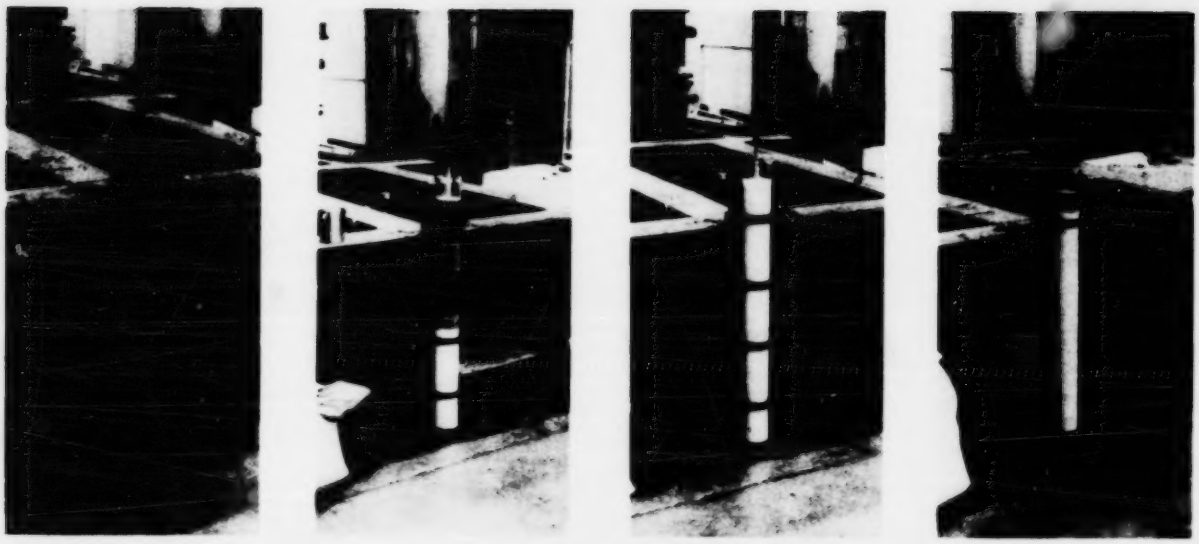


a) Original Layout



b) Recent Layout

FIGURE 5
PARTICULATE COMPACTION TEST STATION



(a) Arrangement Before Pouring

(b) Tube Half Filled with Particulate

(c) Tube Filled with Particulate Before Vibratory Compaction

(d) After Vibratory Compaction

FIGURE 6

VIBRATORY COMPACTION TEST

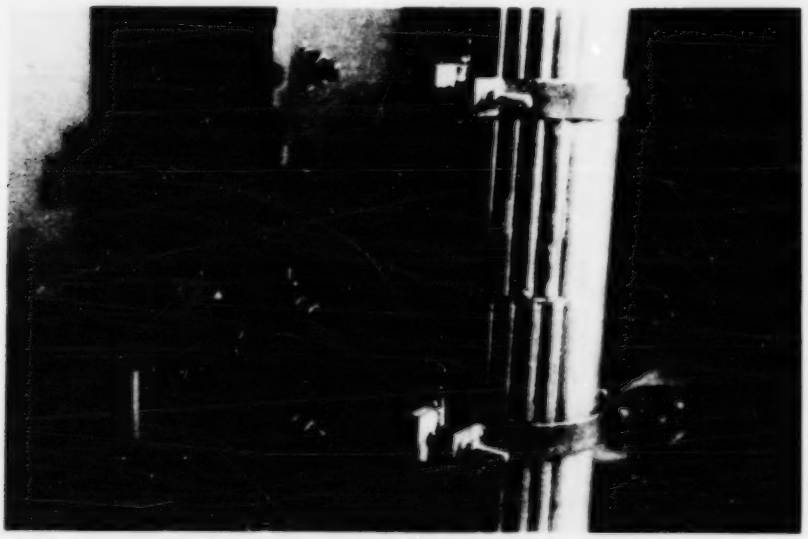
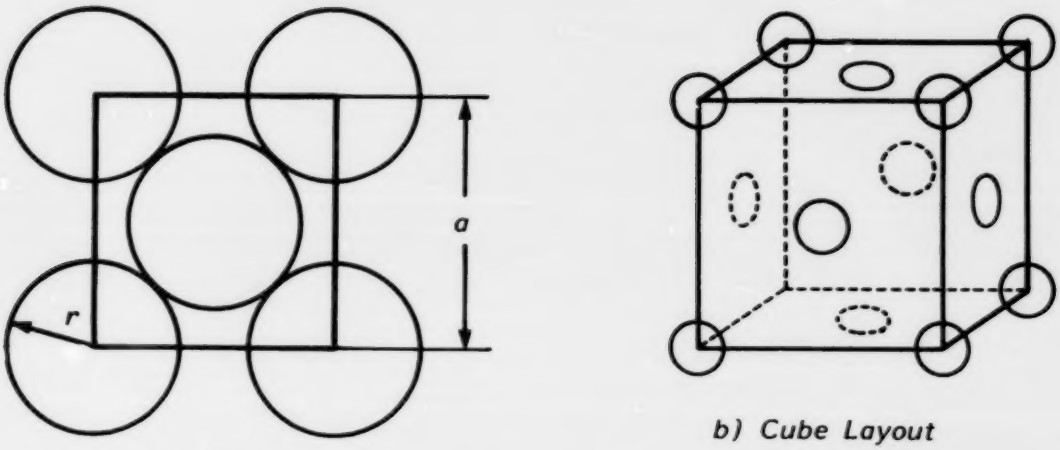


FIGURE 7

ORIENTATION OF BUNDLES



(a) Typical Face

b) Cube Layout

FIGURE 8
BEAD LAYOUT IN FACE-CENTRE-CUBIC ORIENTATION

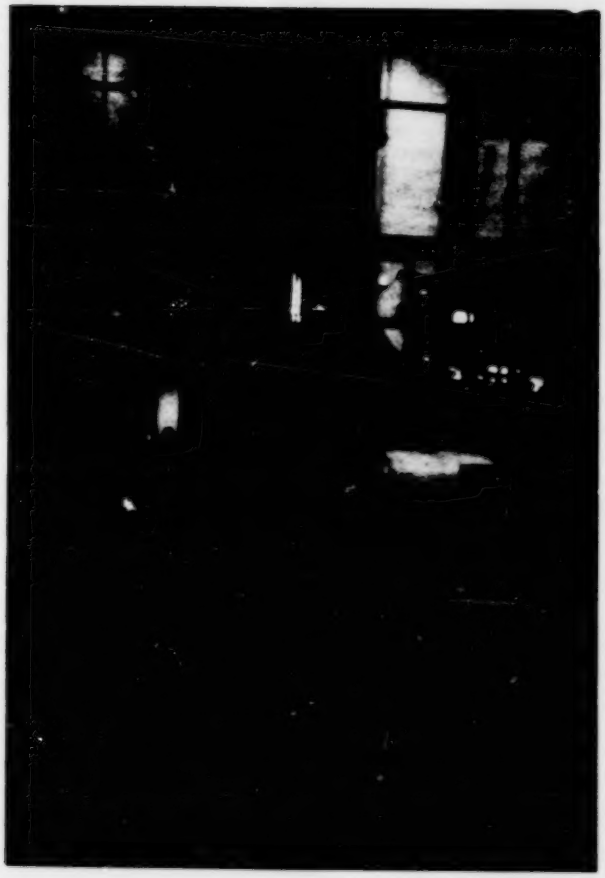


FIGURE 9
VOIDS DURING ZIRCOX TEST

FIGURE 10
SLOPE OF PARTICULATE SURFACE

FIGURE 11
COMPRESSION TEST EQUIPMENT

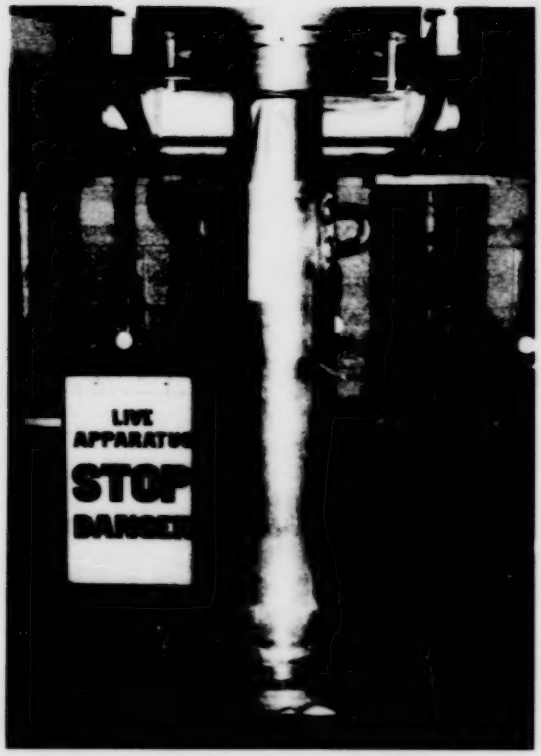


FIGURE 12
COMPRESSION TEST EQUIPMENT
FOR PARTICULATE WITH BUNDLE

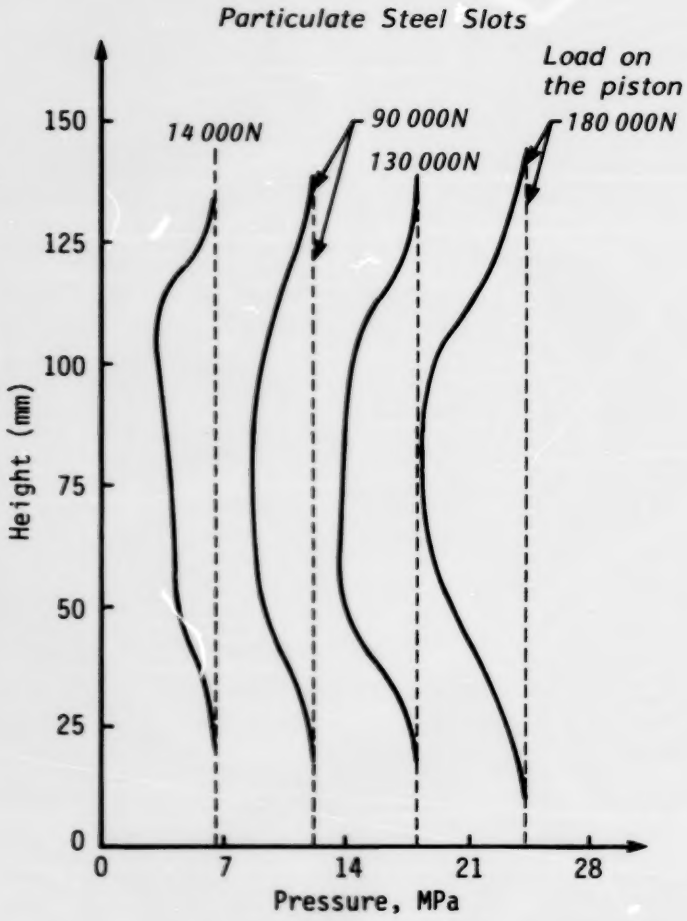
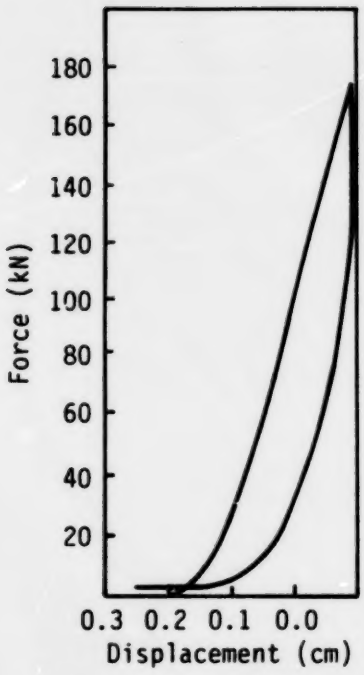
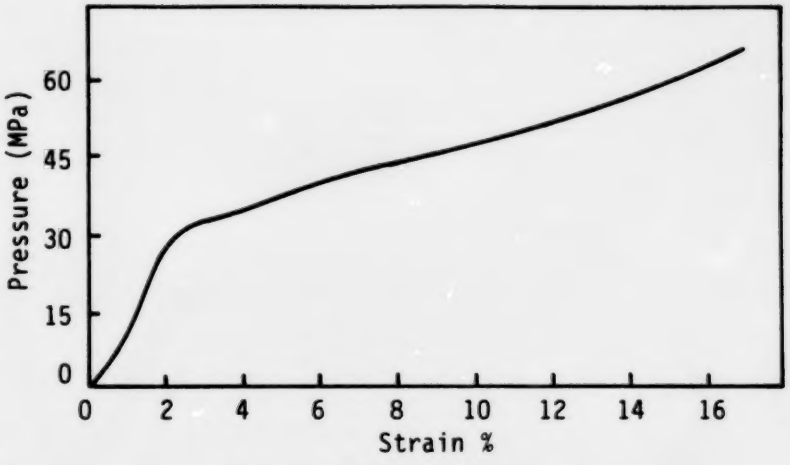


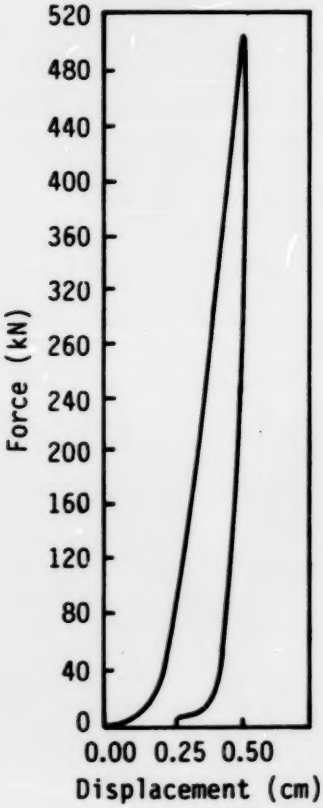
FIGURE 13
AXIAL PRESSURE VARIATION IN SHORT
CYLINDER UNDER FOUR LOADINGS



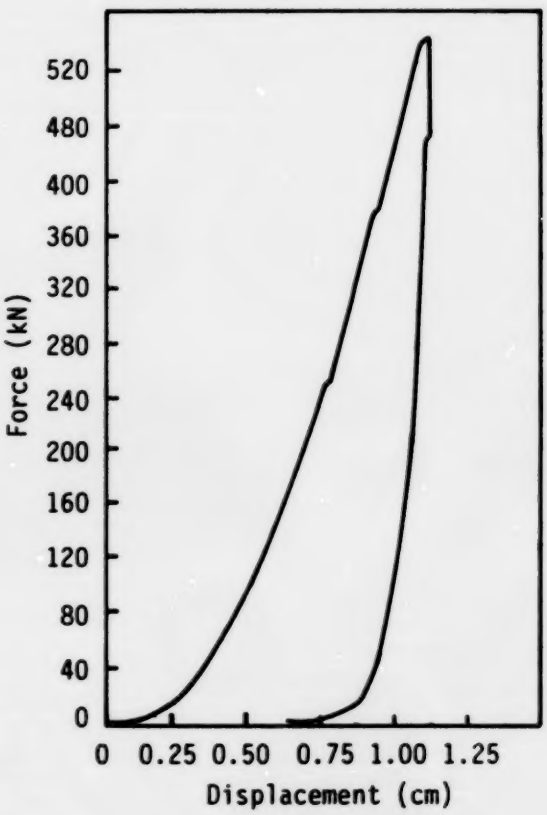
a) Glass beads - low pressure



b) Glass beads - high pressure



c) Sintered bauxite



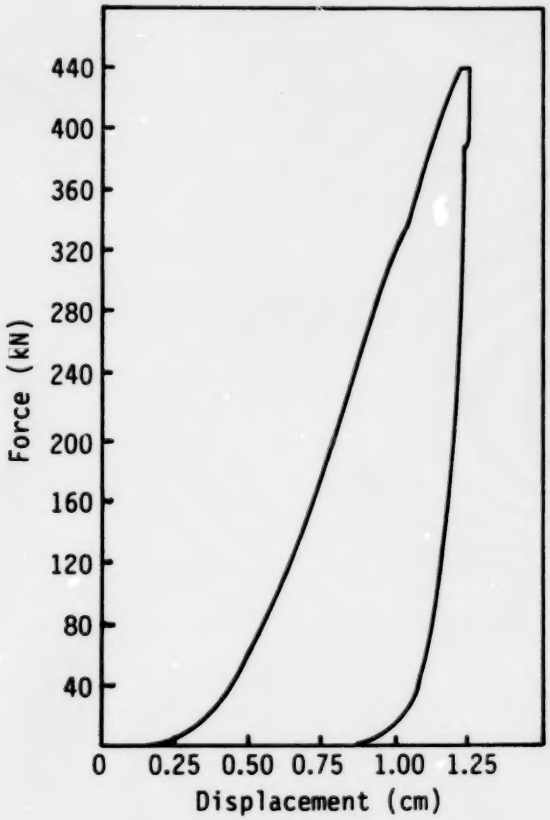
d) Interprop

Note:

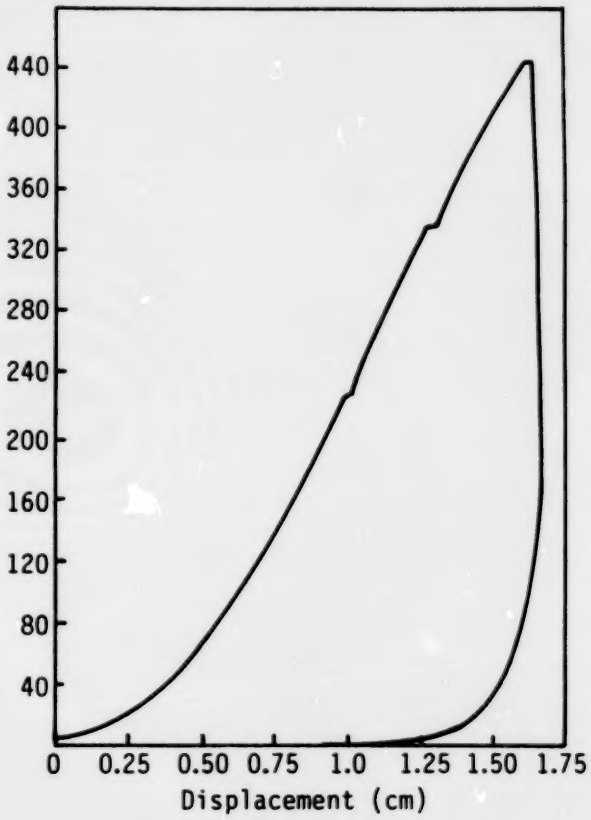
1000m Water head corresponds to the force of 0.092 kN for the cylinder area of 91.6 cm^2

FIGURE 14

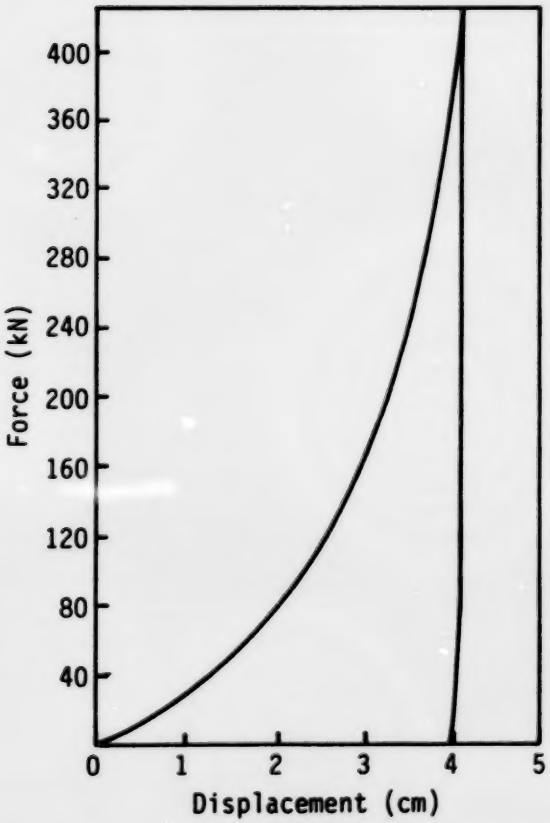
STRENGTH OF PARTICULATE



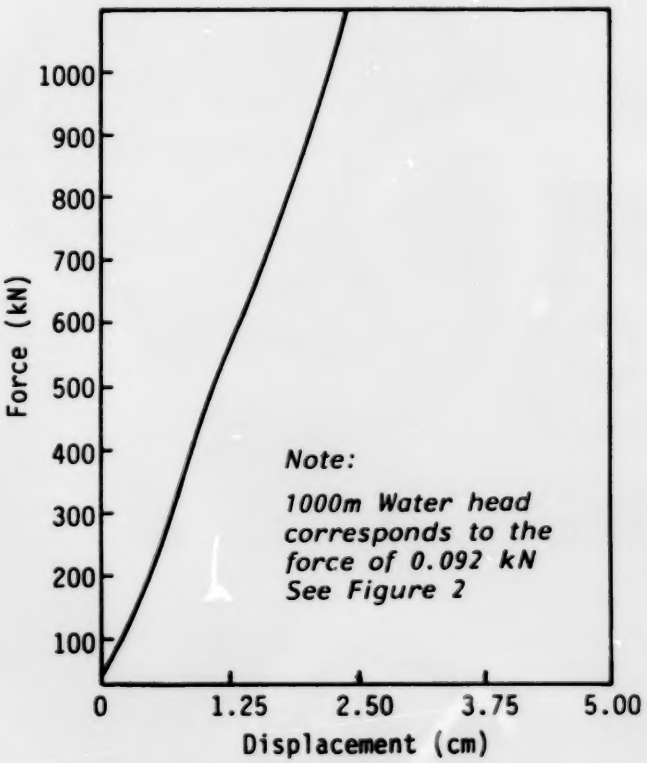
e) Zircon



f) Rutile



g) Zircox 51702

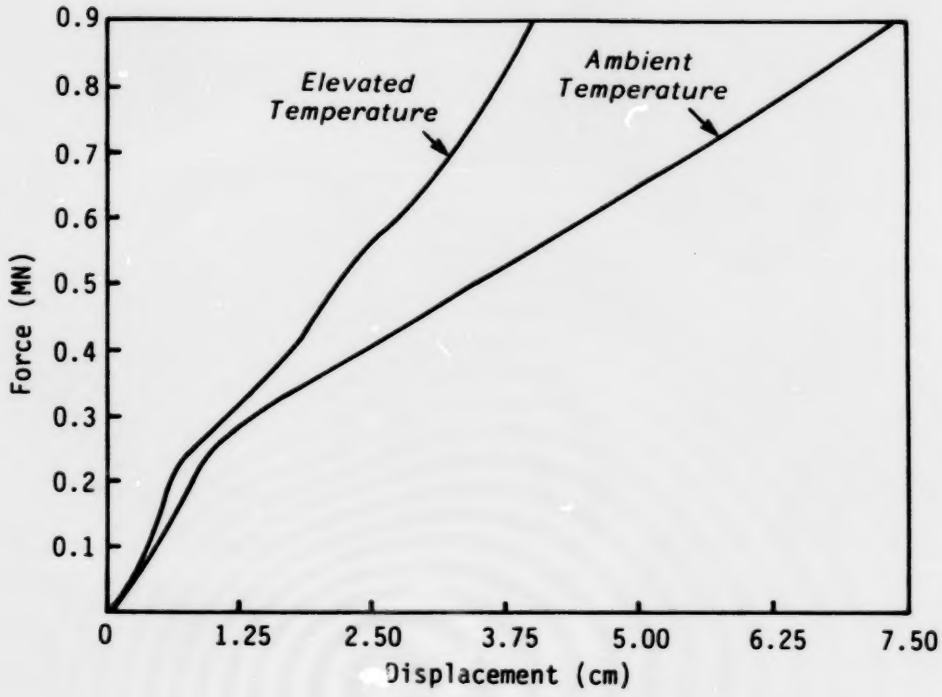


h) Sand

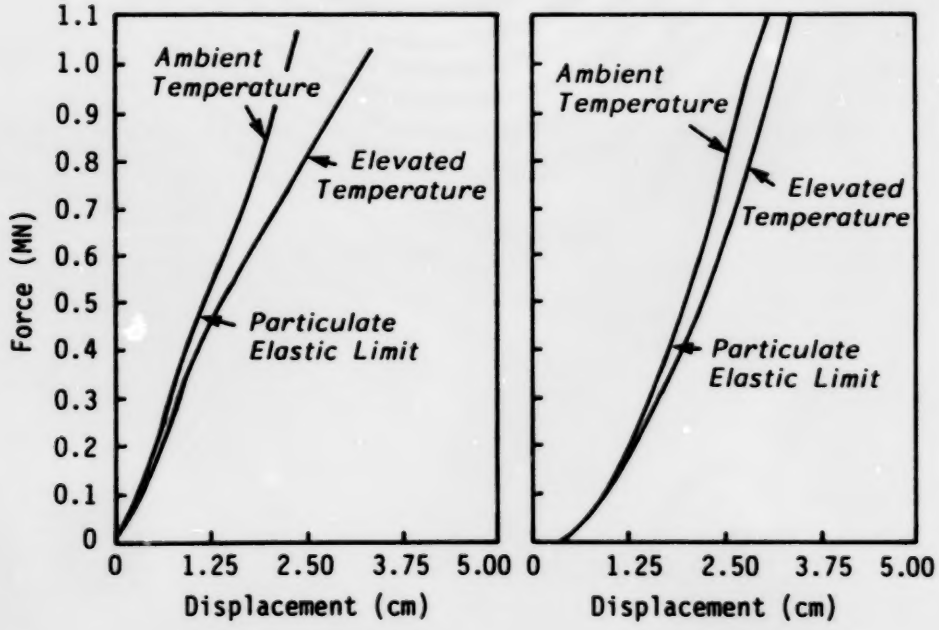
FIGURE 14 (Cont'd)

STRENGTH OF PARTICULATE

172908 RD



a) Glass beads



b) Sand *c) Fine Glass beads*

FIGURE 15

COMPRESSION TESTS AT ELEVATED TEMPERATURE

Note: 1000m water head corresponds to the force of 0.092 MN

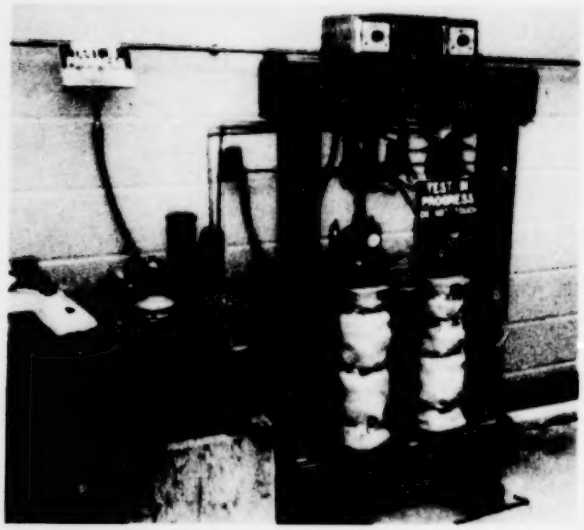


FIGURE 16
CREEP TEST STATION

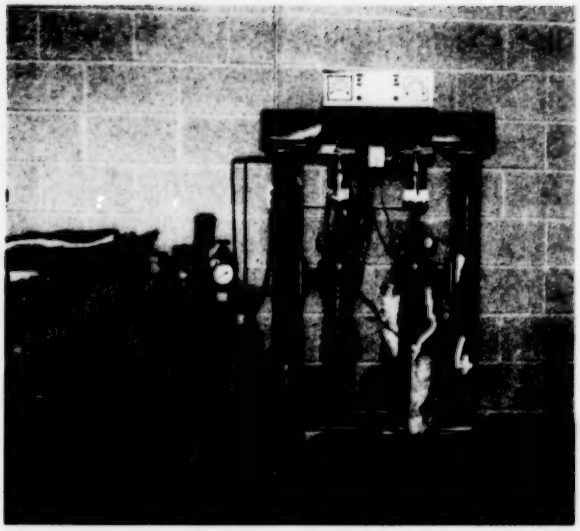


FIGURE 17
STRAIN-GAUGE LOCATIONS

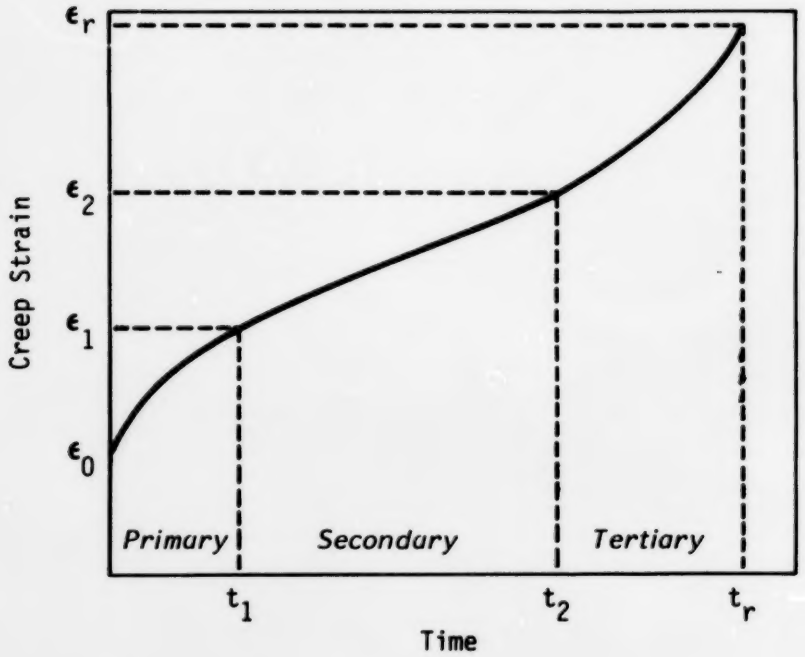
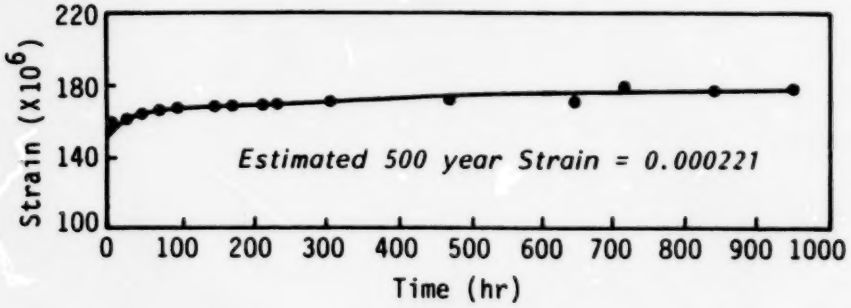
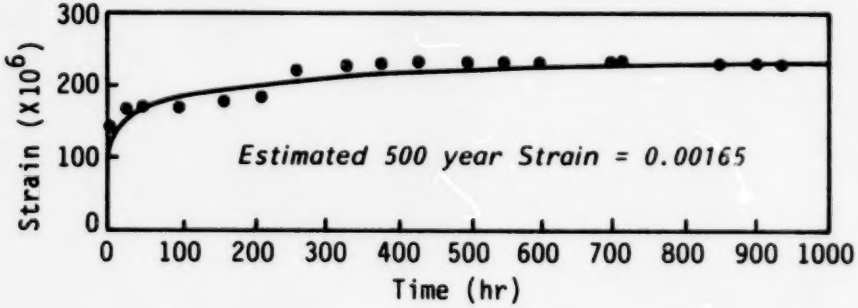


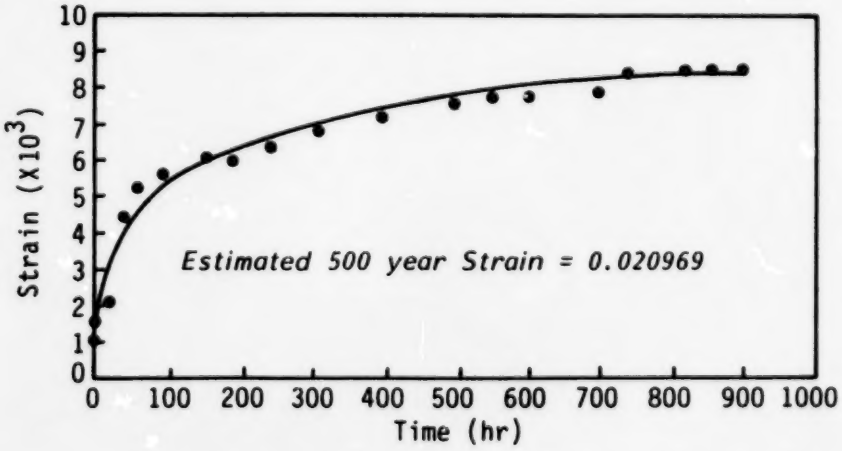
FIGURE 18
SCHEMATIC REPRESENTATION OF A
CREEP-RUPTURE CURVE



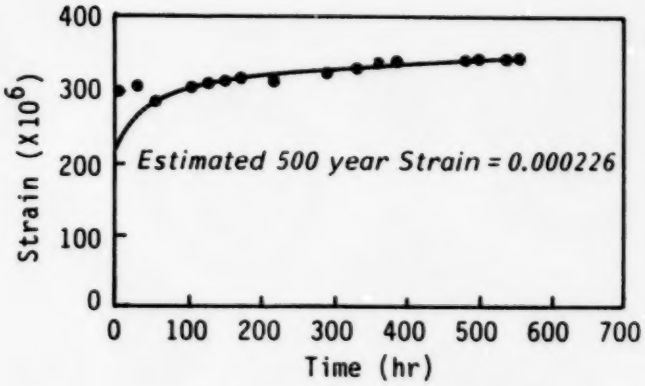
a) Creep in Sand



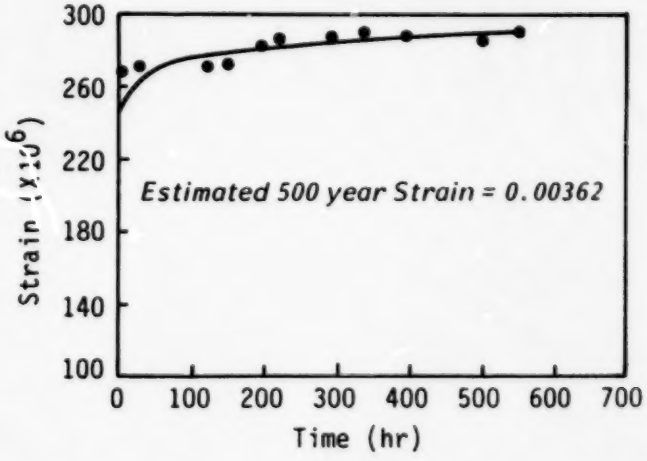
c) Rutile - Zircon - Garnet Combination



e) Zircox 51702



b) Zircon



d) Rutile

FIGURE 19

CREEP TEST RESULTS
MATCHING LOGARITHMIC ASSUMPTION

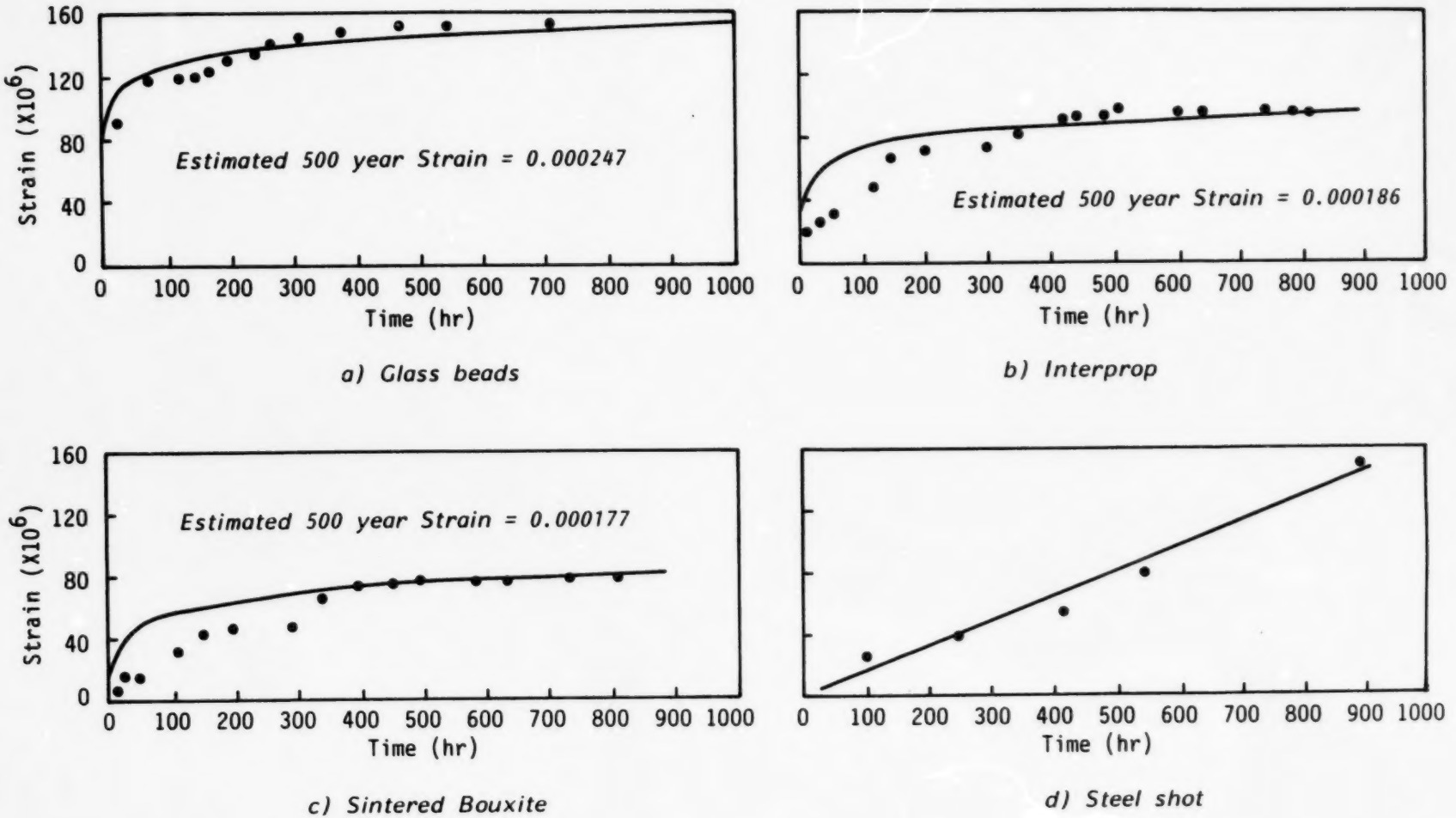
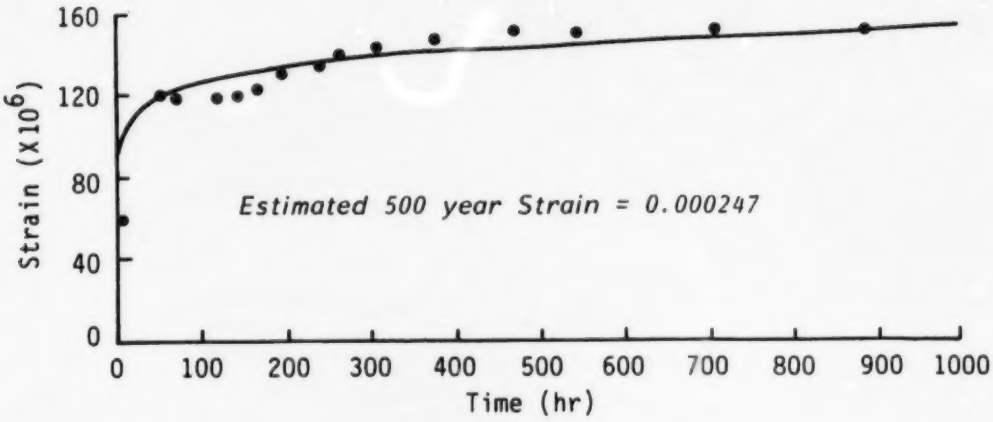
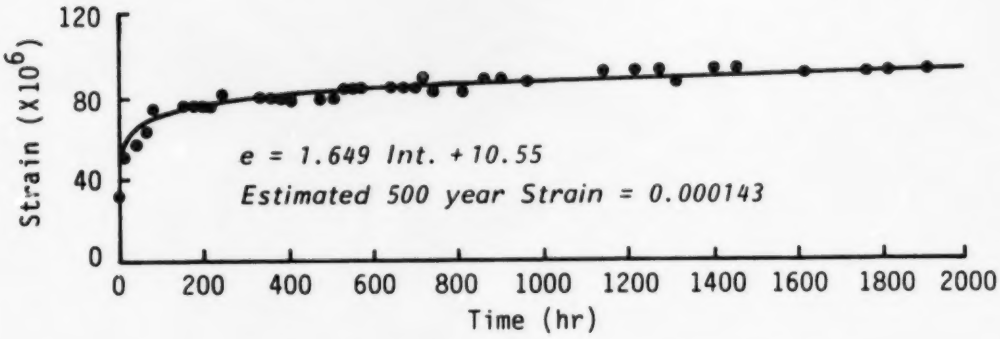


FIGURE 20

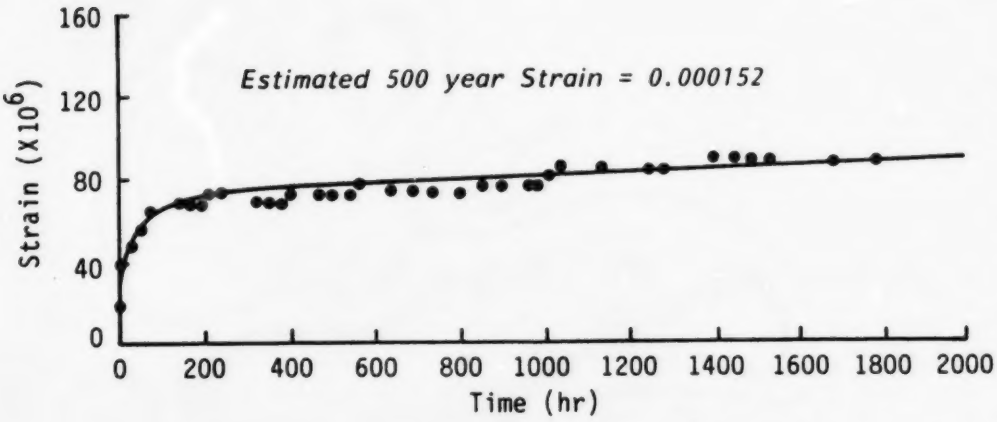
CREEP TEST RESULTS
DEVIATING FROM LOGARITHMIC ASSUMPTION



a) Creep in Glass Beads



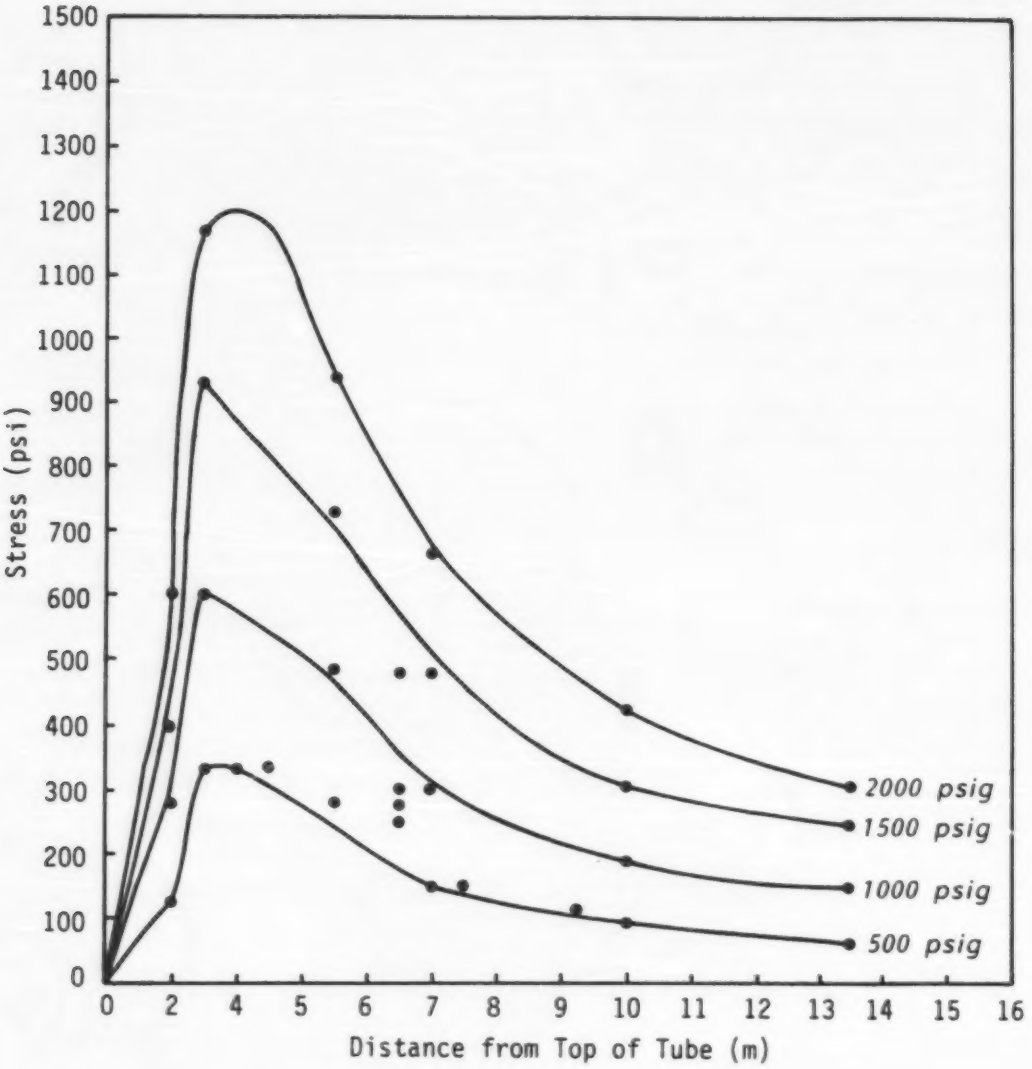
b) Creep of Glass Beads in Di-ionized Water



c) Creep of Glass Beads in Standard Canadian Shield Saline Solution

FIGURE 21

CREEP TEST RESULTS FOR GLASS BEADS



Test of Wedron Sand

FIGURE 22

HOOP STRESS VERSUS DISTANCE FROM TOP OF TUBE

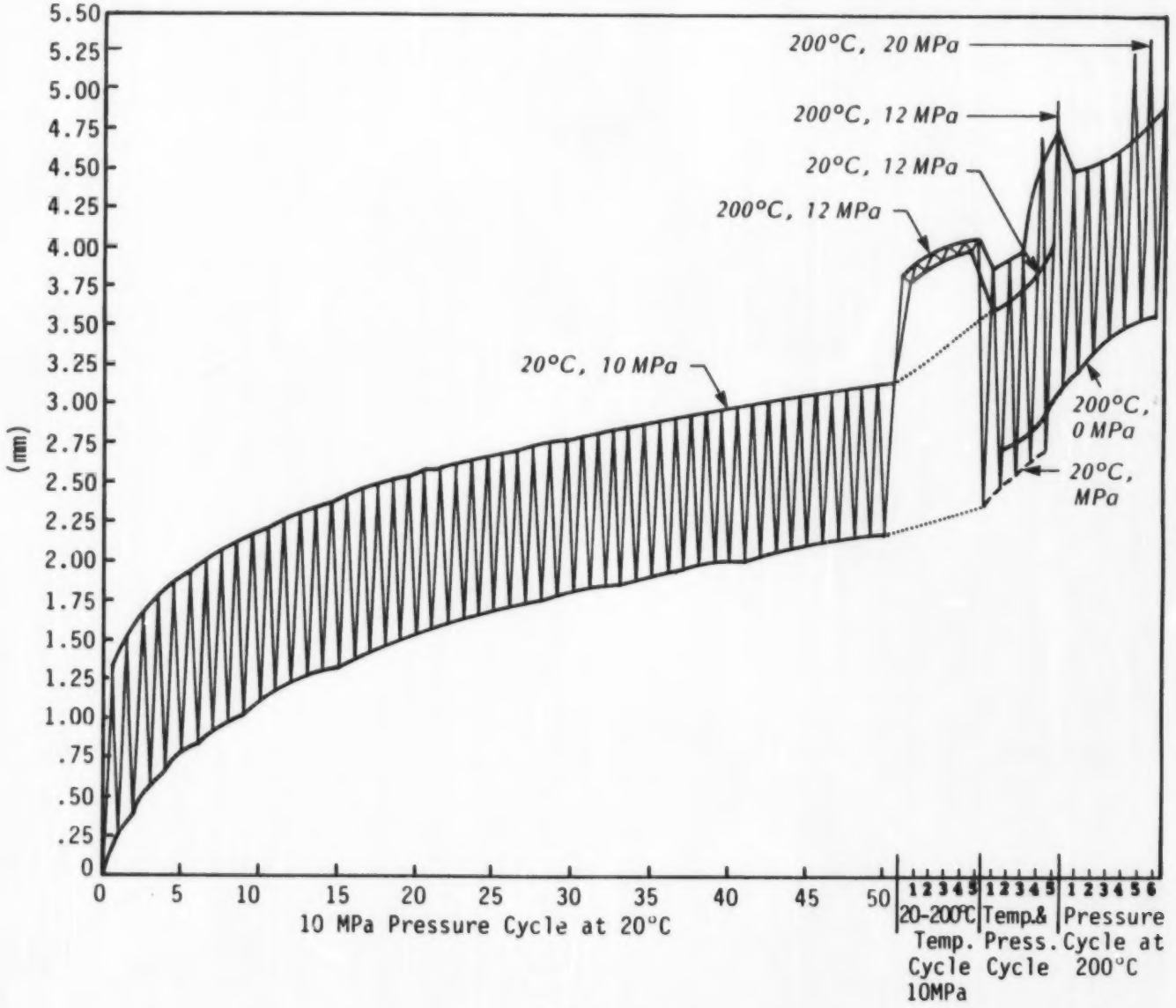


FIGURE 23

PRESSURE AND TEMPERATURE CYCLIC TESTING OF GLASS BEADS

APPENDIX A

PARTICULATE PROPERTIES CALCULATIONS

INTRODUCTION

The compression test program, discussed in Section 7, was conducted to determine the mechanical properties of particulates, such as:

- Young's modulus of elasticity
- Poisson's ratio
- shear modulus of elasticity
- bulk modulus of elasticity.

The equipment used during the test program together with the test procedure, objectives and results were described in Section 7. From the test program we obtained:

- height of particulate H
- force applied to particulate P
- total displacement of the two pistons H
- hoop and axial stresses in the cylindrical shell.

NOMENCLATURE

A	- crossectional area of particulate
Ac	- crossectional arear of cylindrical shell
B	- stiffness coefficient $B = E / [(1 + \nu)(1 - 2\nu)]$
E	- Young's modulus of elasticity
e	- strain
H	- height of particulate column before compression
K	- bulk modulus of elasticity
P	- force acting on particulate
R	- mean radius of the cylinder
r	- inside radius of the cylinder
S	- stress
t	- cylinder's wall thickness
a	- axial direction in cylinder only
c	- circumferential direction in cylinder only
r	- radial direction in cylinder only
x,y,z	- cartesian coordinates used in particulate
ν	- Poisson's ratio
ΔH	- reduction in height of particulate column due to compression

ASSUMPTIONS

The calculations are only approximate and are based on assumptions which may introduce some error.

1. The cylindrical shell is thick and it will not allow particulate to deform in the two mutually perpendicular radial directions (see Figure A-1 for coordinate system convention):

$$e_x = e_y = 0$$

2. The axial strain is uniform, independent of height and is equal to

$$e_z = \Delta H/H$$

3. Axial and hoop stresses in cylindrical shell are averaged through height and assumed to be constant and independent of height.

4. Axial stress inparticulate is equal to the difference between the applied pressure (P/A) and load carried by the cylindrical shell (Sc Ac / A).

$$S_z = \frac{P}{A} - \frac{Sc\ Ac}{A}$$

5. Stresses in particulate are constant throughout each elevation, although they vary with height. This stress is directly related to the hoop stress in the cylinder. Cylinder's wall is thick.

$$S_x = \frac{Sc\ t\ R}{r^2}$$

6. Within the constraints of the cylinder, the particulate is assumed to act like a solid body and equations of the theory of elasticity apply.

$$Sx = B\ [(\ 1 - \nu\ )\ e_x + \nu e_y + \nu e_z]$$
$$Sy = B\ [(\ 1 - \nu\ )\ e_y + \nu e_z + \nu e_x]$$
$$Sz = B\ [(\ 1 - \nu\ )\ e_z + \nu e_x + \nu e_y]$$

RESULTS

By substituting the equations in assumptions 1 through 5 into 6, we obtain equations for the required parameters in form:

Poisson's ratio

$$\nu = \frac{Sc\ R\ t}{Sz\ r + Sc\ R\ t}$$

Young's modulus of elasticity

$$E = Sz\ (H\ /\ \Delta H)\ [1 - 2\nu^2/(1 - \nu)]$$

Shear modulus of elasticity

$$G = E\ /\ 2\ (1 + \nu)$$

Bulk modulus of elasticity

$$K = E/\ 3\ (1 - 2\nu)$$

The above equations were used to derive the values listed in Table II.

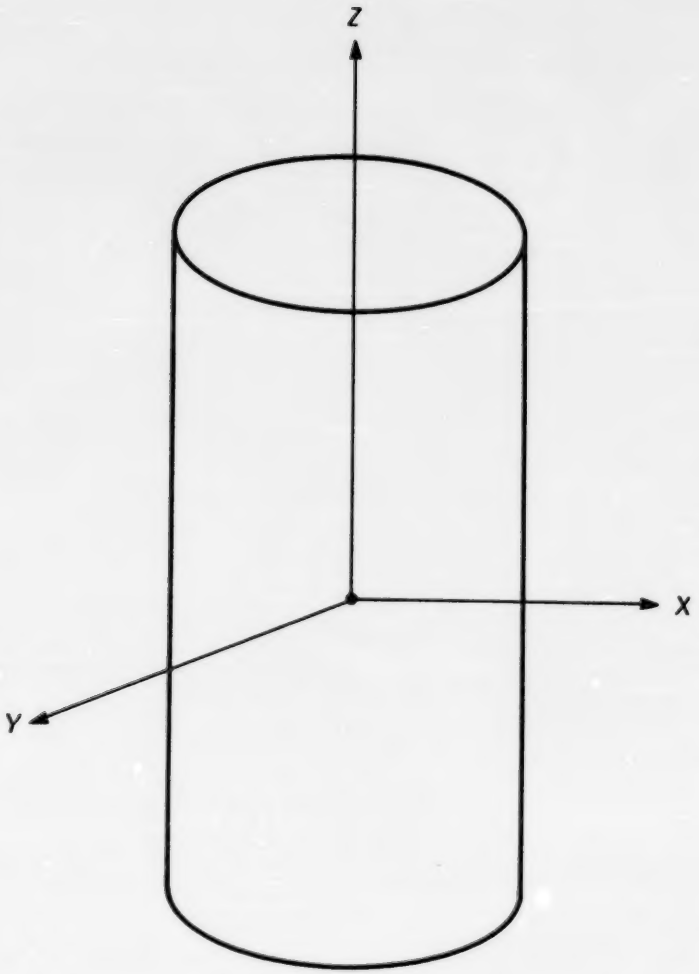


FIGURE A-1
COORDINATE SYSTEM IN PARTICULATE

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